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THE COMPOSITION OF PRECIPITATED AUSTENITE IN 5.5 NI STEEL.(U)

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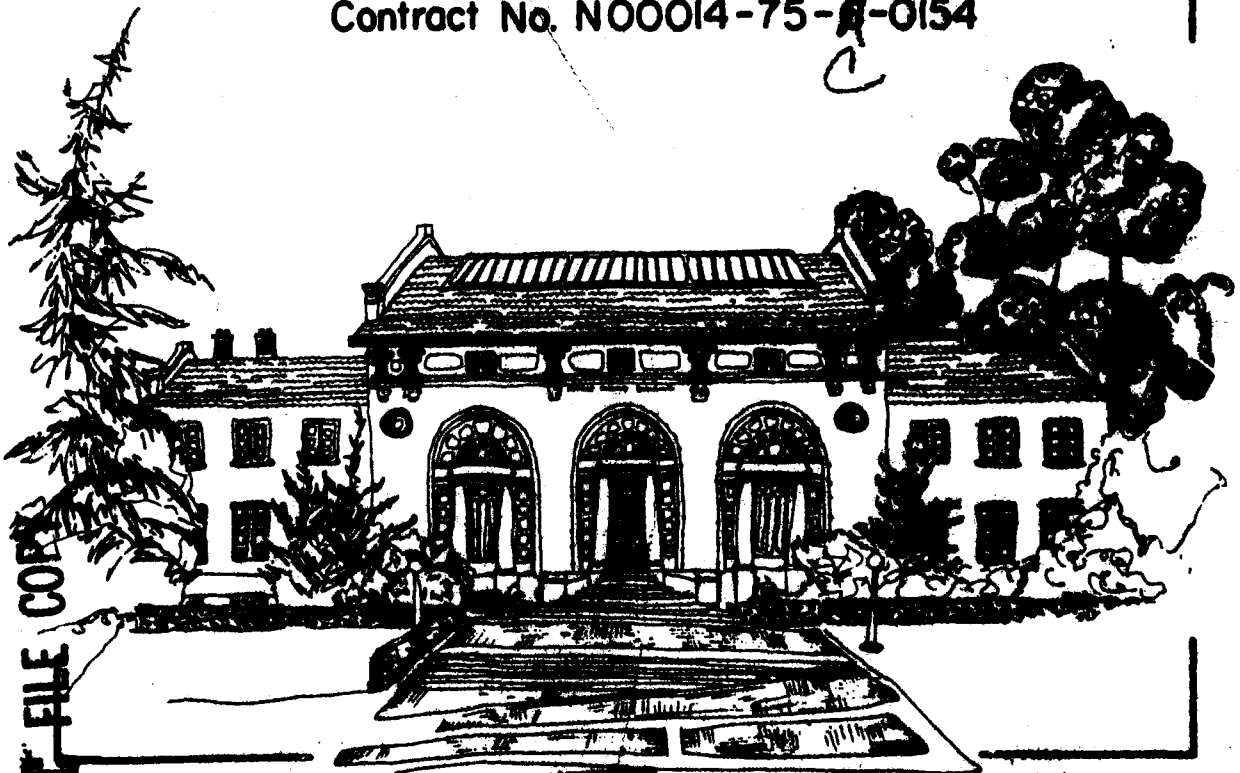
By

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Both Scanning Transmission electron microscopic (STEM) and chemical extrac- tion techniques were used to analyze the chemical content of precipitated austenite in 5.5Ni steel as a function of heat treatment. Austenite was introduced into the steel by tempering at 600°C for 2 hours (OT 1) or 100 hours (OT100), by tempering for 1 hour at 670°C (OL), or by double temper- ing 1 hour at 670°C + 1 hour at 600°C (OLT). The two methods of chemical analysis employed for the analysis of this austenite differ quantitatively (cont.)		

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in the measured austenite composition but are in qualitative agreement. they show an austenite enrichment in Ni, Cr, Mn, and Mo which is most pronounced (and nearly equivalent) for the  $\sigma_{T_{100}}$  and OLT treatments. The similar enrichment of the  $\sigma_{T_{100}}$  and OLT material is interpreted in light of the sequence of reactions leading to the OLT structure. A good correlation is found between the apparent solute enrichment of the precipitated austenite and its thermal stability on cooling to 77°K.

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## INTRODUCTION

The ferritic Fe-Ni steels which have been developed for structural use at cryogenic temperatures are typically given a heat treatment which finishes with an intercritical temper at a relatively low temperature in the two-phase ( $\alpha+\gamma$ ) region<sup>1-6</sup>. During the intercritical temper a fine admixture of austenite phase is precipitated within the prior martensite matrix. If the tempering temperature is sufficiently low and the tempering time sufficiently long, a substantial fraction of the precipitated austenite is retained after cooling to room temperature and remains present on subsequent cooling to service temperatures as low as liquid nitrogen temperature (77°K).

The final intercritical temper is used to lower the ductile-brittle transition temperature of Fe-Ni steels so that good cryogenic structural properties are obtained. Prior research<sup>1-10</sup> suggests that the retained austenite plays a direct role in establishing good low temperature toughness. The toughening mechanism is apparently a combination of the gettering action of the austenite for deleterious species such as carbon<sup>6</sup> and the grain-refining action of the austenite in disrupting the crystallographic alignment of martensite packets<sup>8</sup>. The effectiveness of the austenite in the latter respect depends on the fineness of its distribution through the parent matrix and also depends on its thermal stability<sup>8</sup>; the available evidence<sup>9</sup> suggests that the austenite must be present in the alloy at the time the load is applied if it is to contribute to cryogenic toughness.

While several metallurgical factors may contribute to the thermal stability of precipitated austenite, including its state of coherence and internal strain, the dominant influence should be its composition. Since the austenite is small in size and finely dispersed through the martensite

matrix, however, its composition is difficult to determine. The only published estimate of the austenite composition in an intercritically tempered cryogenic steel known to us is that by Segawa et al.<sup>11</sup>, who used chemical etching techniques to extract retained austenite from a commercial grade 9Ni steel in the quench and tempered condition. These results indicate an enrichment of the austenite in Ni and Mn which is both consistent with the equilibrium phase diagram and with the observed retention of austenite after cooling to liquid nitrogen temperature.

Given this background, the present work was undertaken to gain additional information on the composition of precipitated austenite in intercritically tempered cryogenic steels and to study the sensitivity of the composition to details of the heat treatment. The steel chosen for this research was a commercial grade Fe-5.5Ni cryogenic steel manufactured by the Nippon Steel Company. The particular choice of research material was motivated by the fact that while commercial 9Ni steel has satisfactory toughness at cryogenic temperatures in the quench-and-tempered (QT) condition, the lower nickel grades require a more complex three-step heat treatment, which in the case of the Nippon 5.5Ni steel is designated the QLT treatment, to achieve good toughness at liquid nitrogen temperature (77°K). It was felt that a determination of austenite composition as a function of heat treatment might shed new light on the need for the intermediate (L) treatment.

The work reported here was a part of a larger investigation on the sources of toughness in Fe-5.5Ni in the QLT condition. The part of that work relating to cryogenic mechanical properties and their microstructural interpretation will be published separately.

## EXPERIMENTAL PROCEDURE

A commercial alloy of nominal composition Fe-5.5Ni was provided by the Nippon Steel Corporation. Specific chemical analysis gave the actual composition: Fe-5.86Ni-1.21Mn-0.20Mo-0.25Si-0.69Cr-0.06C-0.008P-0.01S. The alloy, which was initially in the form of a 1.25 in. (3.2 cm) thick plate, was annealed at 1200°C for two hours to remove the effects of prior thermo-mechanical treatment. The plate was then cut into specimens which were given one of the four heat treatments diagrammed in Fig. 1.

Following heat treatment, the fraction of retained austenite in each sample was determined both by x-ray diffraction and by Mössbauer spectroscopy. X-ray diffraction specimens were cut along the longitudinal direction of the plate, then polished and chemically thinned in a solution of 100 ml H<sub>2</sub>O<sub>2</sub> + 3 ml HF for 20 minutes to remove the mechanical effect of the cut surface. The specimens were scanned in a Picker x-ray diffractometer using CuK<sub>α</sub> radiation. The calculation of the volume fraction of the precipitated austenite was based on Miller's method<sup>12</sup>, comparing average integrated intensities of the (220) and (311) austenite peaks to that of the (211) martensite peak. The Mössbauer spectroscopic analysis was accomplished by comparing integrated intensities of austenite and martensite peaks in back-scatter Mössbauer spectra, using the facilities and analysis techniques described by Fultz<sup>13</sup>.

The morphology and distribution of the retained austenite were studied through transmission electron microscopy, using thin foils prepared as described below. To determine the thermal stability of the precipitated austenite, the samples were cooled in liquid nitrogen and their residual austenite contents were measured.

The chemical composition of the retained austenite was measured in two independent ways: through scanning transmission electron microscopy and through chemical extraction.

A. Scanning Transmission Electron Microscopy (STEM).

To prepare foils for transmission electron microscopy, thin slices ~0.3 mm thick were cut from the treated specimens. These were chemically thinned to ~0.15 mm thickness in a solution of 3 ml HF + 100 ml H<sub>2</sub>O<sub>2</sub>, spark cut into 3 mm discs, and sanded down to ~0.05 mm. The thin foils were finally electropolished in a twin jet apparatus at room temperature using a solution of 400 ml CH<sub>3</sub>COOH, 75 g CrO<sub>3</sub> and 21 ml H<sub>2</sub>O and a voltage of 35-45 volts. The foils were studied in a Phillips 400 scanning transmission electron microscope operated at 120 KV.

Chemical analysis of elements of atomic number greater than 11 was accomplished through use of an EDAX unit attached to the microscope. The x-ray count rate data for each element was converted to weight percent concentration using a method suggested by Russ and Duncomb<sup>14</sup>.

The Russ-Duncomb procedure begins from the assumption that the ratio of weight fractions of any two elements in the sample is proportional to the ratio of their integrated x-ray intensities:

$$C_i/C_j = k_{ij} (N_{pi}/N_{pj}), \quad (1)$$

where  $c_i$  is the weight fraction of component  $i$ ,  $N_{pi}$  is its integrated x-ray intensity, and  $k_{ij}$  is a constant:

$$k_{ij} = F_i/F_j \quad (2)$$

where

$$F_i = Q_i W_i T_i / A_i, \quad (3)$$

with  $Q_i$  the ionization cross-section,  $W_i$  the fluorescent yield,  $T_i$  the detector efficiency, and  $A_i$  the atomic weight. The  $F_i$ , and hence the  $k_{ij}$ , can be calculated theoretically as described by Russ & Duncomb<sup>14</sup> or, alternatively, the  $k_{ij}$  may be found directly through measurements on a standard specimen. Given the  $k_{ij}$  for a particular choice of the reference element  $j$  (which is most conveniently taken to be the solvent; Fe in our case), the chemical composition of the sample can be found from equation (1) together with the normalization condition

$$\sum_{i=1}^j c_i = 1 - c', \quad (4)$$

where  $c'$  is the (known or estimated) weight fraction of species outside the range of analysis. In our case,  $c'$  is the total concentration of light elements (<1%) plus the concentration of Mo, which has no fluorescent peaks in the range of analysis.

In the present work, the coefficients  $k_{ij}$  relative to Fe were found theoretically using the method and data reported by Russ and Duncomb<sup>14</sup>, and were tested by determining the composition of the matrix of a quenched foil for comparison with the results of independent chemical analysis. The values of  $k_{ij}$  are tabulated in Table I, which also includes the comparison between alloy composition determined from STEM analysis and that found by bulk analytic techniques. The STEM analysis is accurate except in the case of chromium, where the estimated composition is ~20% too high. This error may be due to fluorescence from the Cr present in the microscope aperture.

### B. Chemical Extraction Analysis

The second method employed for determination of the austenite composition was quantitative analysis of the chemically extracted austenite phase.

To extract the retained austenite phase, the heat-treated specimens were surface machined to remove any oxide film and were then immersed in a solution of 12 wt.% sulfuric acid floated on glycerine<sup>15</sup>. The small (<1 $\mu$ m) islands of austenite are released as the ferrite matrix dissolves in acid and sink into the glycerine, preventing their spontaneous oxidation. Following extraction, the residues were filtered, dried, and examined for crystal structure using both x-ray and backscatter Mössbauer spectroscopy. Both techniques indicated a 100% FCC structure; no residual ferrite was found. The chemical composition of the extracted powder was then determined by wet chemical analysis.

## RESULTS

The microstructure and retained austenite distribution of the Fe-5.5Ni steel is shown as a function of heat treatment in Fig. 2. The austenite volume fractions are given as a function of heat treatment in Table II, both after cooling to room temperature (the condition shown in Fig. 2) and after subsequent refrigeration in liquid nitrogen. The chemical composition of the austenite, as determined by STEM analysis, is given in Table III. Each data point in this table is an average of five separate measurements taken on distinct austenite particles. Since the distribution of austenite compositions is also important, the individual measurements are presented in the extended Table IV.

The composition of the ferrite matrix is also of interest. However, it must be kept in mind that the ferrite matrix will generally consist

of islands having at least two distinct compositions, one of which corresponds to the portion of the ferrite matrix which was tempered during heat treatment without transformation to the austenite phase, and the other of which consists of material transformed to austenite and then retransformed during cooling. Excepting the QLT treatment, in which the double tempering treatment introduces a special complexity, the different regions of the ferrite matrix can usually be distinguished on morphological grounds as illustrated in Fig. 3. The tempered ferrite contains a relatively low dislocation density and shows a polygnization of the residual dislocation structure. The retransformed austenite is fresh martensite having a high internal dislocation density and a lath-like or banded appearance. Table V contains STEM analyses of the composition of the tempered ferrite matrix. Table VI shows the composition of the retransformed austenite after the QL treatment, and compares it to that of the ferrite matrix and the retained austenite phase. An examination of the data in Tables V and VI shows that both the Mn and Cr contents measured in the ferrite phase are, except in the case of the QT<sub>100</sub> treatment, too high to be consistent with the measured composition of the austenite phase and the known composition of the base alloy. The reason for these high values is unknown. some possible explanations are discussed below in the following section.

The chemical composition of the austenite as found through chemical extraction analysis is presented in Table VII. Also included in the table is the weight fraction of the original sample extracted by chemical dissolution. Since the density of the ferrite and austenite phases are very nearly the same, this weight fraction is approximately equal to the volume fraction of the extracted material. The extracted material was found in all cases to be fully austenitic, but was also in all cases significantly less

in quantity than the amount which quantitative x-ray or Mössbauer analysis indicates was present in the original sample. The wet chemical analysis of the residue determined the content of every major constituent of the parent material with the exception of carbon. Carbon analysis was not possible because of carbon contamination of the residue during the extraction process.

#### DISCUSSION

The two chemical analysis techniques employed here, scanning transmission electron microscopy and chemical extraction analysis, differ quantitatively in the retained austenite compositions they reveal. On the other hand, the two techniques are in qualitative agreement both with respect to the accumulation of alloying species within the austenite and the variation of the austenite composition with heat treatment.

##### A. The discrepancy between STEM and Chemical Extraction Results.

While the research reported here cannot fully resolve the quantitative discrepancy between the STEM and chemical extraction results, it is our belief that the STEM analysis is the more accurate of the two. This tentative conclusion is supported by three pieces of evidence: the good agreement between the STEM analysis and the chemical analysis of the parent alloy as presented in Table I, the internal consistency of the STEM data, as given in Table IV, and the quantitative discrepancy between the fraction of chemically extracted product and the volume fraction of austenite in the heat-treated specimens, as revealed in Table VII.

There are at least two reasons why the relatively low fraction of the chemically extracted product may cause the austenite to appear relatively rich in alloying species. First, it is possible that only that fraction of the austenite which is most highly alloyed remains undissolved. This

interpretation is, however, made implausible by the fact that the residue fraction is in all cases more than one-half of the total fraction of austenite but has a measured composition which is in all cases richer in alloying elements than any of the five separate austenite particles analysed by STEM for each heat treatment. A second and more plausible explanation concerns the possible leaching of iron from the austenite produced during dissolution, a process which would simultaneously increase the apparent alloy content of the austenite phase and decrease the weight fraction of the residue.

The quantitative accuracy of the STEM data is, however, also open to question, particularly in light of the anomalously high Mn and Cr contents shown in Tables V and VI. The reason for those anomalously high compositions is unknown. Possible causes include extra fluorescence from the aperture of the microscope or contaminants within the microscope, sample preparation procedure which may result in enrichment of Mn and Cr or preferential presentation of regions rich in Mn and Cr, and inherent limitations of the technique itself.

#### B. The Composition of the Precipitated Austenite

Both STEM and extraction analyses indicate that the precipitated austenite is enriched in the alloy elements Ni, Mn, and Cr. The chemical extraction analysis also suggests an enrichment in Mo. The degree of enrichment is a strong function of heat treatment. In all cases, however, the nickel content of the austenite remains substantially below the value which would be inferred from the binary Fe-Ni phase diagram and well below the 16-18% Ni content found by Segawa et al.<sup>11</sup> through extractive chemical analysis of the retained austenite in 9Ni steel. This low Ni content presumably reflects the thermodynamic influence of co-segregation of the other

alloying species and, particularly in the case of the QT<sub>2</sub> treatment, the kinetic limitation on Ni segregation.

The importance of kinetics in limiting the Ni content of the austenite is anticipated from the extremely low diffusivity of Ni in Fe. Bulk diffusivity data taken from binary Fe-Ni alloys<sup>16</sup> gives a mean diffusion length  $\bar{x} = \sqrt{2Dt}$  of only  $\sim 0.01 \mu\text{m}$  in  $\alpha$ -iron after 1 hr at 600°C. It is hence questionable whether even a 100-hr treatment at this temperature is sufficient to establish complete equilibrium in a structure containing austenite islands  $\sim 1 \mu\text{m}$  apart, unless dislocation "pipe" or surface diffusion plays a significant role. The diffusivities of Mn and Cr are approximately an order of magnitude higher than that of Ni in  $\alpha$ -iron at 600°C, hence these species are expected to segregate in advance of Ni or to relax with its changing composition profile.

While the composition of the retained austenite is sensitive to heat treatment, the data presented in Table IV suggests that the composition does not vary substantially from particle to particle over the local region of the alloy available for analysis. The particle-to-particle variation found in STEM analysis appears to be due primarily to the limited accuracy of the technique itself<sup>17</sup>.

#### C. The Dependence of Composition on Heat Treatment

The change in austenite composition with heat treatment may be plausibly interpreted given the low diffusivity of substitutional species, particularly nickel. The austenite formed on two hours tempering at 600°C (QT<sub>2</sub>) is low in nickel content but becomes progressively richer in nickel as the tempering time is increased, reaching  $\sim 9.1\%$  after 100 hours (QT<sub>100</sub>). The Mn and Cr contents also increase with tempering time.

If the tempering temperature is raised to 670°C, however, Ni diffusion is enhanced (by roughly an order of magnitude) and a more extensive precipitation of austenite simultaneously decreases the austenite inter-particle distance. As a consequence even one hour of tempering time (QL) is sufficient to establish a Ni content of ~8.4% in the austenite and to raise the Mn and Cr contents to values nearly equal to those found after 100 hours at 600°C. If the QL specimen is then given a second 1 hour temper at 600°C, the QLT treatment, the Ni content of the austenite rises further to approach the value obtained after 100 hours at 600°C. Both STEM and extraction analyses show a concomitant rise in the contents of Mn, Cr, and Ni; the extraction analysis suggests a further enrichment in Mo. Interestingly, both STEM and extraction analysis reveal a Cr content after the QLT treatment which is nearly twice that found after 100 hours at 600°C, and the extraction analysis suggests a similar near doubling of the Mo content.

The rapid adjustment of austenite composition during the final 1 hour 600°C temper in the QLT treatment appears to be a result of the mechanism of formation of this retained austenite phase. A morphological analysis of the resulting microstructure, illustrated in Fig. 4, suggests that the austenite particles retained after the final temper form within islands of enriched composition left over from the prior 670°C treatment. Since those islands, which are either retained austenite or fresh martensite in the QL microstructure, already contain a high concentration of Ni, Mn, Cr, and Mo, their decomposition can produce particles of high-alloy austenite with a minimal need for long-range diffusion. In effect, the alloy behaves, locally, as if it were an 8-9% Ni steel rather than a 5% Ni steel.

#### D. Thermal Stability of Precipitated Austenite

The retention of austenite after refrigeration in liquid nitrogen is given as a function of heat treatment in Table II. A comparison of this data to the chemical composition measurements presented in Tables III and VII shows a clear correlation between the thermal stability of the austenite and its chemical composition. The austenite introduced by the QT<sub>2</sub> and QL treatments, which is relatively lean in solute species, is also relatively unstable. The austenite produced in the QT<sub>100</sub> and QLT treatments is relatively rich in alloying species and is largely retained on cooling to 77°K.

The very great difference between the thermal stabilities of the austenite produced by the QL and QLT treatments raises some questions since the compositional difference between these austenites is, while significant, not substantial. The greater stability of the QLT product may indicate the importance of the secondary alloying elements Cr, Mo, and Mn, or the influence of austenite morphology. It should be kept in mind, moreover, that the carbon content of the austenite could not be measured in this work, though difference in carbon content will certainly affect austenite stability.

#### CONCLUSION

While the quantitative precision of the chemical analyses of precipitated austenite reported here is uncertain, the qualitative trends revealed in the variation of composition with heat treatment are both consistent and plausible. They indicate a co-segregation of Ni, Mn, Cr, and Mo, and the reveal a gradual enrichment in nickel after long-time tempering at 600°C. The results suggest that a double-tempering treatment, the QLT treatment, which involves a one-hour temper at 600°C, is successful in enhancing the Ni content of austenite precipitated at 600°K. The thermal stability of the

austenite on cooling to 77°K is found to increase with its alloy content. The QLT treatment hence appears to be a useful technique for achieving a highly-alloyed, stable austenite in reasonable heat treatment time. Its success appears to result from the mode of austenite precipitation: the final austenite forms within regions previously enriched by segregation during the high temperature intermediate temper.

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Figure 1. Schematic diagram of heat treatments.

Figure 2. Transmission electron micrographs of (a)  $QT_2$ , (b)  $QT_{100}$ , (c) QL and (d) QLT. "A" denotes retained austenite.

Figure 3. Transmission electron micrograph of QL-treated specimen. A, M and F denote retained austenite phase, fresh martensite phase transformed from reverted austenite and ferrite phase respectively.

Figure 4. Transmission electron micrograph of QLT-treated specimen. (a) Bright field image and (b) dark field image of retained austenite.

Table I. Chemical composition

Wt. %	Fe	C	Mn	Si	P	S	Ni	Cr	Mo
Wet Analysis	Bal	0.063	1.21	0.25	0.008	0.01	5.86	0.69	0.20
STEM	Bal	-	1.13	0.29	-	-	5.88	0.88	-
Analysis	(1)		(0.96)	(0.51)			(1.17)	(0.91)	

( ): kij value with Fe as reference element j.

TABLE II. Volume percent of precipitated austenite.

	At 298°K	After 77°K Refrigeration
QT <sub>2</sub>	5.9	3.8
QT <sub>100</sub>	8.2	7.9
QL	8.8	2.0
QLT	9.2	8.8

TABLE III. Chemical composition of the precipitated austenite (by STEM).

	QT <sub>2</sub>	QT <sub>100</sub>	QL	QLT
Fe	89.5	85.0	86.1	82.8
Ni	6.6	9.1	8.4	8.8
Mn	2.2	3.8	3.7	4.3
Cr	1.2	1.5	1.3	3.2
Si	0.1	0.2	0.1	0.5

Total: 99.5% in weight

TABLE IV. Chemical composition of each island of retained austenite detected by STEM given in wt. %.

		Fe	Ni	Mn	Cr	Si
QT <sub>2</sub>	1	90.0	6.3	1.9	1.2	0.1
	2	88.2	7.7	2.6	0.9	0.1
	3	88.8	6.9	2.4	1.3	0.1
	4	90.6	5.4	2.1	1.3	0.1
	5	89.5	6.5	1.3	1.3	0.1
QT <sub>100</sub>	1	84.4	10.2	3.3	1.4	0.2
	2	85.0	8.5	4.0	1.8	0.2
	3	85.1	8.8	3.9	1.5	0.2
	4	85.0	9.2	4.0	1.1	0.1
	5	85.8	8.5	3.5	1.7	0.2
QL	1	86.9	8.6	2.8	1.1	0.1
	2	85.5	8.4	4.3	1.3	0.1
	3	84.3	9.3	4.1	1.7	0.1
	4	87.3	7.1	3.7	1.3	0.1
	5	86.3	8.6	3.3	1.2	0.1
QLT	1	82.6	9.5	3.7	3.3	0.5
	2	83.4	9.1	2.9	2.9	0.9
	3	81.2	8.7	6.2	3.2	0.4
	4	84.4	7.8	4.2	2.8	0.4
	5	82.8	8.9	3.8	3.6	0.5

TABLE V. Chemical composition of ferrite matrix (STEM) in wt.%.

	QT <sub>2</sub>	QT <sub>100</sub>	QL	QLT
Fe	91.8	94.2	93.2	91.8
Ni	5.4	4.1	4.2	4.8
Mn	1.3	0.7	1.2	1.2
Cr	1.1	0.6	1.1	1.4
Si	0.2	0.2	0.1	0.6

TABLE VI. Chemical composition of each phase in QL condition (STEM) given in wt.%.

	Fe	Ni	Mn	Cr	Si
$\alpha$ -ferrite	93.2	4.2	1.2	1.1	0.1
$\alpha'$ -martensite	86.7	8.2	3.4	1.1	0.1
$\gamma$ -austenite	86.1	8.4	3.7	1.3	0.1

TABLE VII. Chemical composition of the powder extracted in  
12% H<sub>2</sub>SO<sub>4</sub> solution

		600°C, 2 hrs.	600°C, 100 hrs.	670°C, 1 hr	670°C, 1 hr + 600°C, 1 hr.
Vol.% of retained austenite		5.9	8.2	8.8	9.2
Wt.% of extracted powder		2.1	5.3	4.3	7.2
Chemical Comp. (wt.%)	Fe	82.24	76.93	79.55	74.81
	Ni	9.61	13.18	10.82	12.50
	Mn	3.99	4.62	4.03	4.10
	Cr	1.52	1.43	1.27	2.57
	Si	0.81	1.20	0.51	1.60
	Mo	1.83	2.64	3.82	4.42

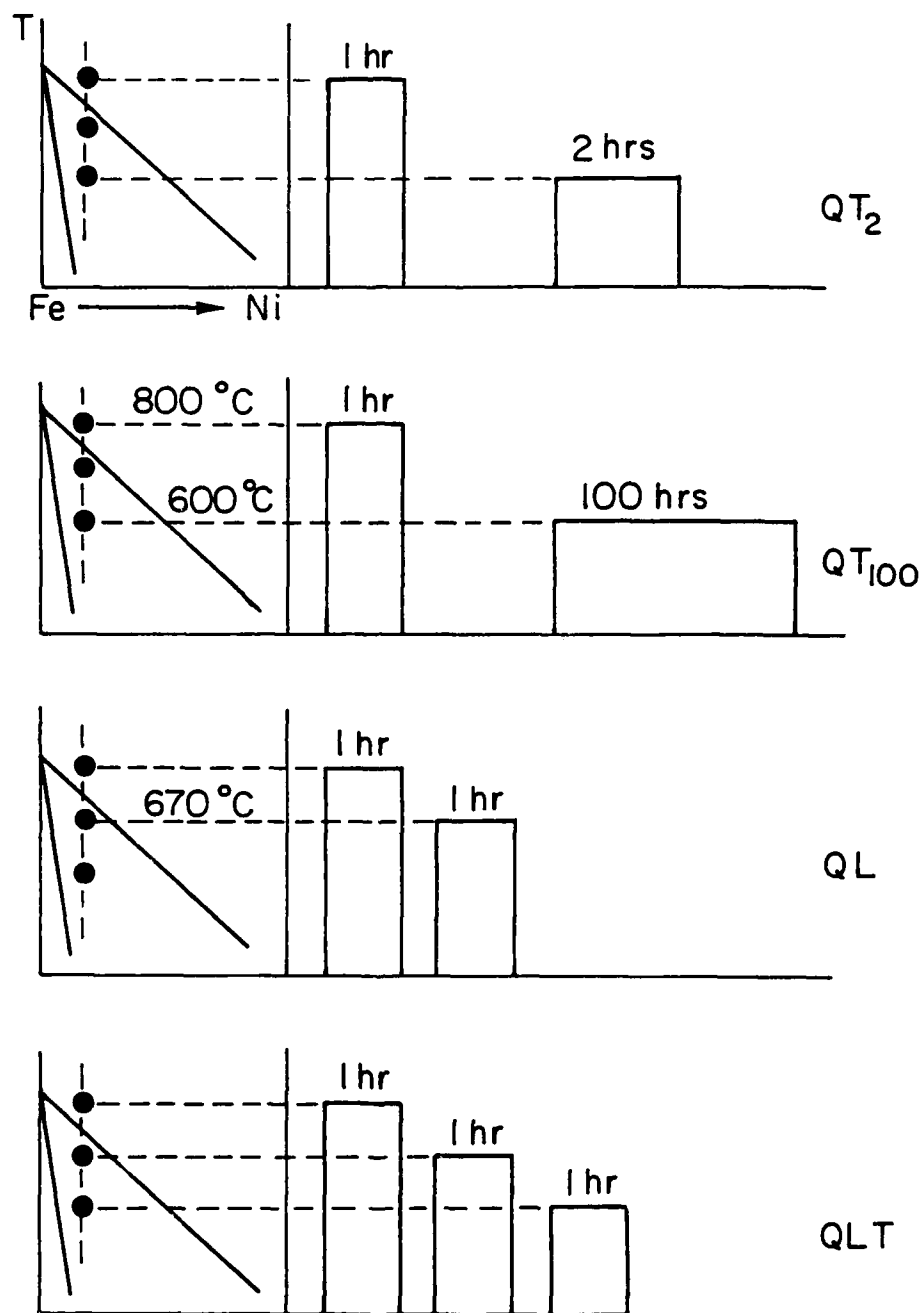


Figure 1

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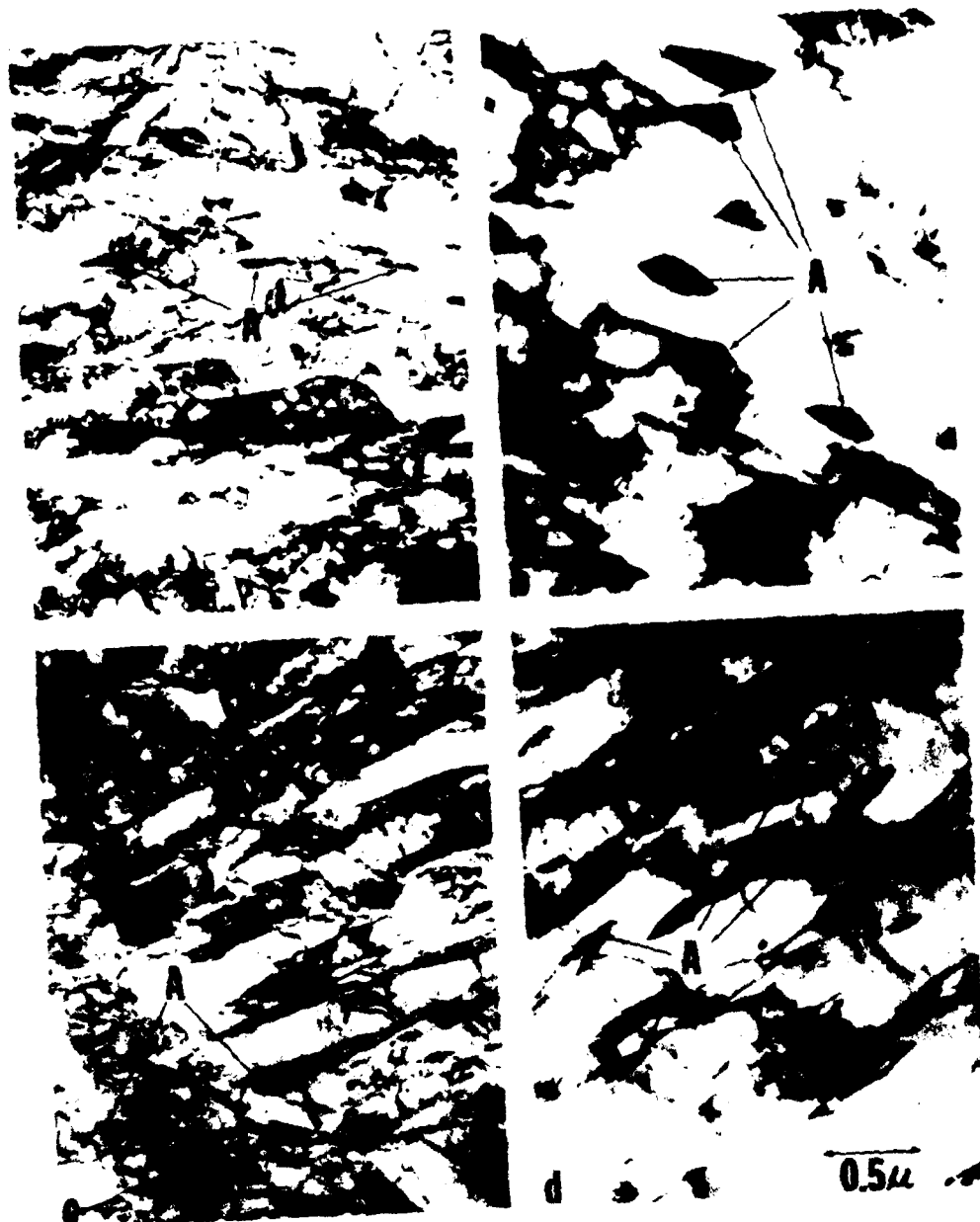


Figure 2

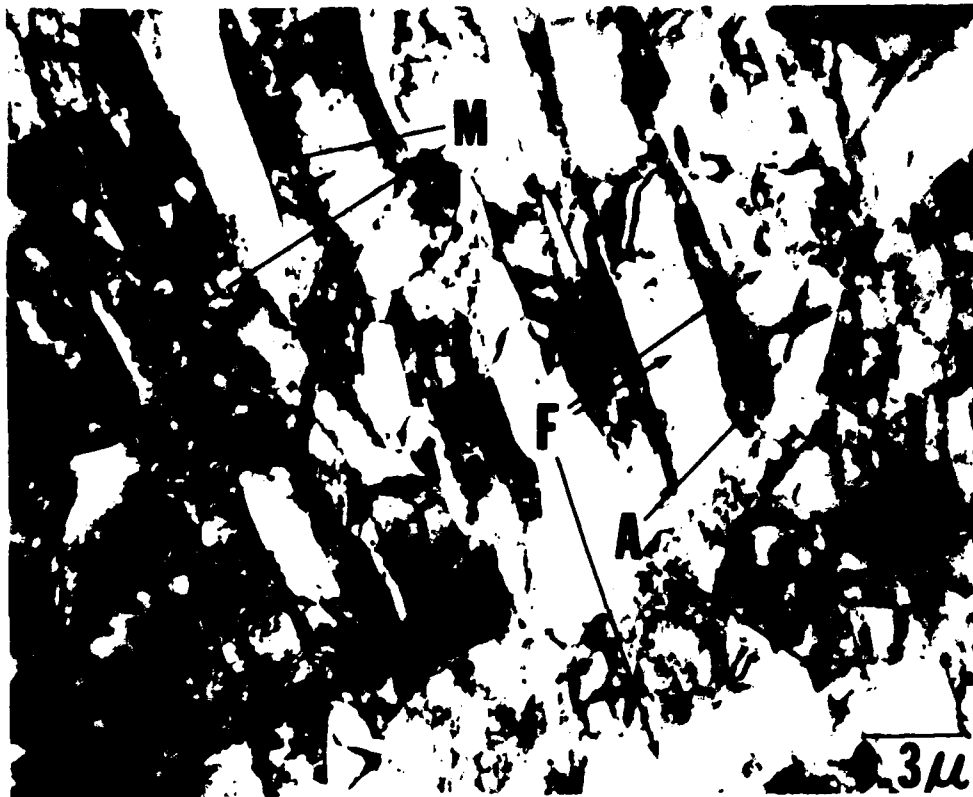


Figure 5

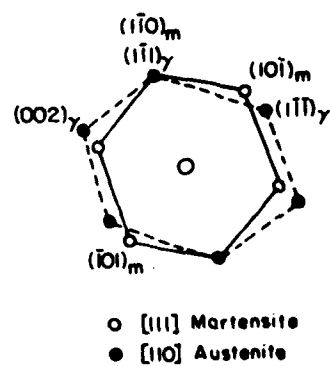
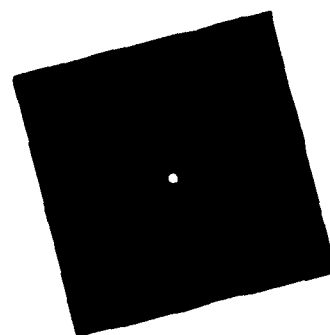
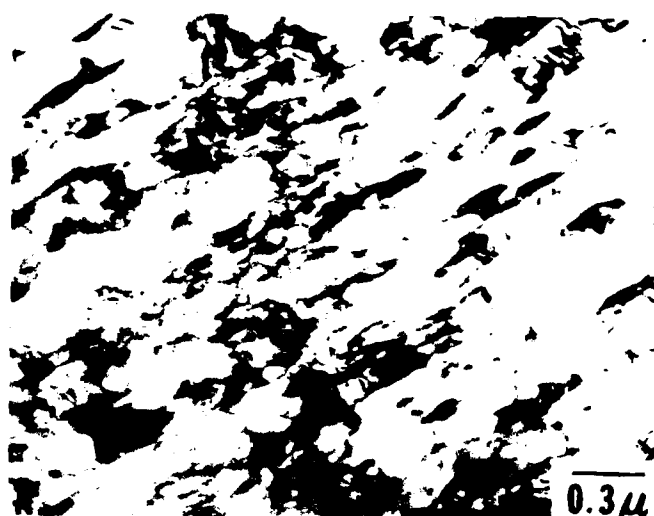


Figure 1